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**(54) Thickening solvents with elastomeric silicone polyethers**

(57) Low molecular weight siloxane fluids are thickened herein by use of silicone elastomers. These elastomers are made by combining in one pot an =Si-H containing siloxane, a mono-alkenyl polyether, an alpha, omega-diene and a low molecular weight siloxane fluid. An elastomer, i.e. gel, with polyether groups is thus produced. The elastomer can also be swollen with low molecular weight siloxane fluid under shear force, to provide a uniform silicone paste. This paste has excellent spreadability upon rubbing, and possesses unique rheological properties by being thixotropic and shear thinning. The silicone paste is easily emulsified with water to form a stable uniform emulsion, without using a surfactant to allow normally immiscible materials to become intimately mixed.

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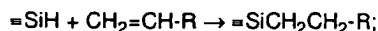
## Description

This invention is directed to thickened low molecular weight siloxane fluids or solvents, in the form of silicone elastomers swollen into silicone pastes, which can subsequently be formed into silicone emulsions.

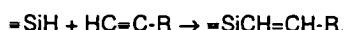
"Cross-links" are junctions of polymer strands in a three-dimensional network. They may be viewed as long-chain branches which are so numerous that a continuous insoluble network or gel is formed.

Increasingly, platinum catalyzed hydrosilylation reactions are being used to form networks. They typically involve reactions between a low molecular weight siloxane containing several  $\text{=Si-H}$  groups, and a high molecular weight siloxane containing several  $\text{=Si-vinyl}$  groups, or vice versa.

Attractive features of this mechanism are that (i) no by-products are formed, (ii) cross-linking sites and hence network architecture are narrowly defined, and (iii) hydrosilylation will proceed even at room temperature to form the networks. In the mechanism, crosslinking involves addition of  $\text{=SiH}$  across double bonds, i.e.,



or crosslinking involves addition of  $\text{=SiH}$  across triple bonds, i.e.,



We have utilized this mechanism, but by employing some unobvious and unique modifications of the mechanism, we have formulated a new range of products having a wide range of properties and applications. In particular, one unique aspect is that our silicone paste is used to form an emulsion without the need of a surfactant. This is of considerable value in the personal care arena where skin sensitivity due to the presence of certain surfactants can be an issue.

Our invention relates to a method of making a silicone elastomer by combining and reacting in one pot (A) an  $\text{=Si-H}$  containing polysiloxane; (B) an unsaturated hydrocarbon such as an alpha, omega-diene; (C) a platinum catalyst; (D) a mono-alkenyl polyether; and (E) a solvent, until a silicone elastomer is formed.

As another feature, additional solvent is added to the silicone elastomer, and the solvent and silicone elastomer are sheared until a silicone paste is formed.

As a further feature, water is added to the silicone paste, and the water and silicone paste are sheared until a silicone emulsion is formed. The product silicone emulsion is formed free of a surfactant.

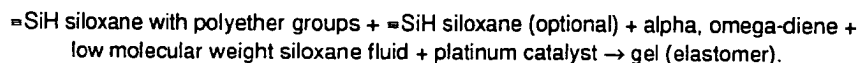
Silicone elastomers, silicone pastes, and silicone emulsions, prepared according to these methods, have particular value and utility in treating hair, skin, or underarm areas of the human body. In addition, these elastomers, pastes and emulsions are capable of forming barrier films after evaporation of any solvent or volatile component.

The invention and relevant steps are performed according to the process illustrated with reference to the below procedure.

## Step 1: Incorporation of polyether



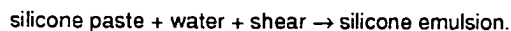
## Step 2: Gelation



## Step 3: Shearing and swelling



## Step 4: Emulsification

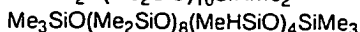


In Step 1, the molar ratio of the polyether to the  $\text{=SiH}$  in the  $\text{=SiH}$  siloxane is between zero and one.

In Step 2, the weight ratio of the low molecular weight siloxane fluid to the weight of the  $\text{=SiH}$  siloxane with polyether groups and the alpha, omega-diene is from 1-98, but preferably is between 3-10. The molar ratio of  $\text{=SiH}$  in the  $\text{=SiH}$  siloxane with polyether groups to vinyl in the alpha, omega-diene is from 20:1 to 1:20, but preferably is 1:1. While

Step 2 can include a mixture of various types of compounds, at least one =SiH containing siloxane must include a polyether group.

For example, one formulation found especially suitable for Step 2 is a mixture containing the following compounds:



1,5-hexadiene, and decamethylcyclopentasiloxane. In these formulas, Me is, and hereafter denotes, methyl and Q is  $-\text{CH}_2\text{CH}_2\text{CH}_2(\text{CH}_2\text{CH}_2\text{O})_{10}\text{H}$ .

In Step 3, the silicone paste contains 80-98 percent by weight of the low molecular weight siloxane fluid or other fluid or solvent to be thickened.

In Step 4, the weight ratio of water to the silicone paste is 95:5 to 5:95.

In the present invention, however, we have eliminated separate Steps 1 and 2 of the prior art, so that all of the reactants (i.e., the =SiH containing siloxane(s), the mono-alkenyl polyether, the alpha, omega-diene, the low molecular weight siloxane or solvent and the platinum catalyst), are all combined and reacted in one pot.

The =Si-H containing polysiloxane is represented by compounds of the formula  $\text{R}_3\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_3$  referred to as type  $\text{A}^1$ , and compounds of the formula  $\text{HR}_2\text{SiO}(\text{R}'_2\text{SiO})_c\text{SiR}_2\text{H}$  or compounds of the formula  $\text{HR}_2\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_2\text{H}$  referred to as type  $\text{A}^2$ . In the above three formulas, R, R' and R'', are alkyl groups with 1-6 carbon atoms; a is 0-250; b is 1-250; and c is 0-250. The molar ratio of compounds  $\text{A}^2:\text{A}^1$  is 0-20, preferably 0-5. In preferred embodiments, compounds of types  $\text{A}^1$  and  $\text{A}^2$  are used in the reaction; however, it is possible to successfully conduct the reaction using only compounds of type  $\text{A}^1$ .

The =Si-H containing polysiloxane  $\text{A}^1$  can also comprise an alkylhydrogen cyclosiloxane or an alkylhydrogen-dialkyl cyclosiloxane copolymer, represented in general by the formula  $(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b$  where R', R'', a and b are as defined above. Preferably, a is 0-7; and b is 3-10. Some representative compounds are  $(\text{OSiMeH})_4$ ,  $(\text{OSiMeH})_3(\text{OSiMeC}_6\text{H}_{13})$ ,  $(\text{OSiMeH})_2(\text{OSiMeC}_6\text{H}_{13})_2$ , and  $(\text{OSiMeH})(\text{OSiMeC}_6\text{H}_{13})_3$ .

The most preferred unsaturated hydrocarbon is an alpha, omega-diene of the formula  $\text{CH}_2=\text{CH}(\text{CH}_2)_x\text{CH}=\text{CH}_2$  where x is 1-20. Some representative examples of suitable alpha, omega-dienes for use herein are 1,4-pentadiene; 1,5-hexadiene; 1,6-heptadiene; 1,7-octadiene; 1,8-nonadiene; 1,9-decadiene; 1,11-dodecadiene; 1,13-tetradecadiene and 1,19-eicosadiene.

However, other unsaturated hydrocarbons can be used such as alpha, omega-diynes of the formula  $\text{CH}(\text{CH}_2)_x\text{C}\equiv\text{CH}$ ; or alpha, omega-ene-yne of the formula  $\text{CH}_2=\text{CH}(\text{CH}_2)_x\text{C}\equiv\text{CH}$  where x is 1-20. Some representative examples of suitable alpha, omega-diynes for use herein are 1,3-butadiyne  $\text{HC}\equiv\text{C}-\text{C}\equiv\text{CH}$  and 1,5-hexadiyne (dipropargyl)  $\text{HC}\equiv\text{C}-\text{CH}_2\text{CH}_2-\text{C}\equiv\text{CH}$ . One representative example of a suitable alpha, omega-ene-yne is hexene-5-yne-1  $\text{CH}_2=\text{CHCH}_2\text{CH}_2\text{C}\equiv\text{CH}$ .

Our reaction requires a catalyst to effect the reaction between the =SiH containing siloxanes, the mono-alkenyl polyether, and the alpha, omega-diene. Suitable catalysts are Group VIII transition metals, i.e., the noble metals. Such noble metal catalysts are described in US Patent 3,923,705 which shows typically platinum catalysts and their preparation. One preferred platinum catalyst is Karstedt's catalyst, which is described in US Patents 3,715,334 and 3,814,730. Karstedt's catalyst is a platinum divinyl tetramethyl disiloxane complex, typically containing one weight percent of platinum, carried in a polydimethyl-siloxane fluid or in a solvent such as toluene.

The particular catalyst used in the examples herein is 20  $\mu\text{l}$ , 40  $\mu\text{l}$  or 200  $\mu\text{l}$  portions of Karstedt catalyst, as 1-2 weight percent of platinum, carried in a two centistoke ( $\text{mm}^2/\text{s}$ ) polydimethylsiloxane fluid, or in a volatile methyl siloxane such as decamethylcyclopentasiloxane. Another preferred platinum catalyst is a reaction product of chloroplatinic acid and an organosilicon compound containing terminal aliphatic unsaturation. It is more fully described in US Patent 3,419,593. The noble metal catalysts are used in amounts from 0.00001-0.5 part platinum per 100 weight parts of =SiH containing polysiloxane, preferably 0.00001-0.02 part, most preferably 0.00001-0.002 part.

The mono-alkenyl polyether is a compound of the formula  $\text{CH}_2=\text{CH}(\text{CH}_2)_x\text{O}(\text{CH}_2\text{CH}_2\text{O})_y(\text{CH}_2\text{CH}_2\text{CH}_2\text{CHO})_z\text{T}$  or a compound of the formula  $\text{CH}_2=\text{CH}-\text{Q}-\text{O}(\text{CH}_2\text{CH}_2\text{O})_y(\text{CH}_2\text{CH}_2\text{CH}_2\text{CHO})_z\text{T}$ . In these formulas, T represents an end group which is hydrogen atom; a  $\text{C}_1$ - $\text{C}_{10}$  alkyl group such as methyl, ethyl, propyl, butyl and decyl; an aryl group such as phenyl; or a  $\text{C}_1$ - $\text{C}_{20}$  acyl group such as acetyl, propionyl, butyryl, lauroyl, myristoyl, and stearoyl. Q is a divalent linking group containing unsaturation such as phenylene  $-\text{C}_6\text{H}_4-$ . The value of x is 1-6; y is zero or a value of 1-100; z is zero or a value of 1-100; with the proviso that y and z cannot both be zero.

Some representative examples of suitable mono-alkenyl polyethers are compounds with the formulas  $\text{CH}_2=\text{CHCH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_7\text{H}$ ,  $\text{CH}_2=\text{CHCH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_{10}[\text{CH}(\text{CH}_3)\text{CH}_2\text{O}]_4\text{H}$ , and  $\text{CH}_2=\text{CHCH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_7\text{C}(\text{O})\text{CH}_3$ .

By "solvent", we mean (i) organic compounds, (ii) compounds containing a silicon atom, (iii) mixtures of organic compounds, (iv) mixtures of compounds containing a silicon atom, or (v) mixtures of organic compounds and compounds containing a silicon atom; used on an industrial scale to dissolve, suspend or change the physical properties of other materials.

Compounds containing a silicon atom are preferably low molecular weight silicone oils. The phrase low molecular weight silicone oil includes compounds containing a silicon atom such as (i) low molecular weight linear and cyclic volatile methyl siloxanes, (ii) low molecular weight linear and cyclic volatile and non-volatile alkyl and aryl siloxanes and (iii) low molecular weight functional linear and cyclic siloxanes. Most preferred, however, are low molecular weight linear and cyclic volatile methyl siloxanes (VMS).

VMS compounds correspond to the average unit formula  $(\text{CH}_3)_a\text{SiO}_{(4-a)/2}$  in which a has an average value of two to three. These compounds contain siloxane units joined by  $\text{Si-O-Si}$  bonds. Representative units are monofunctional "M" units  $(\text{CH}_3)_3\text{SiO}_{1/2}$  and difunctional "D" units  $(\text{CH}_3)_2\text{SiO}_{2/2}$ .

The presence of trifunctional "T" units  $\text{CH}_3\text{SiO}_{3/2}$  results in the formation of branched linear or cyclic volatile methyl siloxanes. The presence of tetrafunctional "Q" units  $\text{SiO}_{4/2}$  results in the formation of branched linear or cyclic volatile methyl siloxanes.

Linear VMS have the formula  $(\text{CH}_3)_3\text{SiO}[(\text{CH}_3)_2\text{SiO}]_y\text{Si}(\text{CH}_3)_3$ . The value of y is 0-5. Cyclic VMS have the formula  $\{(\text{CH}_3)_2\text{SiO}\}_z$ . The value of z is 3-9. Preferably, these volatile methyl siloxane have boiling points less than 250°C. and viscosities of 0.65 to 5.0 centistokes ( $\text{mm}^2/\text{s}$ ).

Representative linear VMS are hexamethyldisiloxane (MM) with a boiling point of 100°C., viscosity of 0.65  $\text{mm}^2/\text{s}$  and formula  $\text{Me}_3\text{SiOSiMe}_3$ ; octamethyltrisiloxane (MDM) with a boiling point of 152°C., viscosity of 1.04  $\text{mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiOMe}_2\text{SiOSiMe}_3$ ; decamethyltetrasiloxane ( $\text{MD}_2\text{M}$ ) with a boiling point of 194°C., viscosity of 1.53  $\text{mm}^2/\text{s}$  and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_2\text{SiMe}_3$ ; dodecamethylpentasiloxane ( $\text{MD}_3\text{M}$ ) with a boiling point of 229°C., viscosity of 2.06  $\text{mm}^2/\text{s}$  and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_3\text{SiMe}_3$ ; tetradecamethylhexa-siloxane ( $\text{MD}_4\text{M}$ ) with a boiling point of 245°C., viscosity of 2.63  $\text{mm}^2/\text{s}$  and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_4\text{SiMe}_3$ ; and hexadecamethylheptasiloxane ( $\text{MD}_5\text{M}$ ) with a boiling point of 270°C., viscosity of 3.24  $\text{mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_5\text{SiMe}_3$ .

Representative cyclic VMS are hexamethylcyclotrisiloxane ( $\text{D}_3$ ) a solid with a boiling point of 134°C. and formula  $\{(\text{Me}_2\text{SiO})\}_3$ ; octamethylcyclotetrasiloxane ( $\text{D}_4$ ) with a boiling point of 176°C., viscosity of 2.3  $\text{mm}^2/\text{s}$ , and formula  $\{(\text{Me}_2\text{SiO})\}_4$ ; decamethylcyclopentasiloxane ( $\text{D}_5$ ) with a boiling point of 210°C., viscosity of 3.87  $\text{mm}^2/\text{s}$ , and formula  $\{(\text{Me}_2\text{SiO})\}_5$ ; and dodecamethylcyclohexasiloxane ( $\text{D}_6$ ) with a boiling point of 245°C., viscosity of 6.62  $\text{mm}^2/\text{s}$ , and formula  $\{(\text{Me}_2\text{SiO})\}_6$ .

Representative branched VMS are heptamethyl-3-((trimethylsilyl)oxy)trisiloxane ( $\text{M}_3\text{T}$ ) with a boiling point of 192°C., viscosity of 1.57  $\text{mm}^2/\text{s}$ , and formula  $\text{C}_{10}\text{H}_{30}\text{O}_3\text{Si}_4$ ; hexamethyl-3,3-bis((trimethylsilyl)oxy)trisiloxane ( $\text{M}_4\text{Q}$ ) with a boiling point of 222°C., viscosity of 2.86  $\text{mm}^2/\text{s}$ , and formula  $\text{C}_{12}\text{H}_{36}\text{O}_4\text{Si}_5$ ; and pentamethyl ((trimethylsilyl)oxy) cyclo-trisiloxane ( $\text{MD}_3$ ) with the formula  $\text{C}_8\text{H}_{24}\text{O}_4\text{Si}_4$ .

Our process also includes using low molecular weight linear and cyclic volatile and non-volatile alkyl and aryl siloxanes represented respectively by formulas  $\text{R}_3\text{SiO}(\text{R}_2\text{SiO})_y\text{SiR}_3$  and  $(\text{R}_2\text{SiO})_z$ . In the latter formulas, R is alkyl groups with 2-20 carbon atoms or aryl groups such as phenyl. The value of y is 0-80, preferably 5-20. The value of z is 3-9, preferably 4-6. These polysiloxanes have viscosities generally in the range of 1-100 centistokes ( $\text{mm}^2/\text{s}$ ). Polysiloxanes can also be used where y has a value sufficient to provide polymers with a viscosity in the range of 100-1,000 centistokes ( $\text{mm}^2/\text{sec}$ ). Typically, y is 80-375. Illustrative of such polysiloxanes are polydimethylsiloxane, polydiethylsiloxane, polymethylethylsiloxane, polymethylphenylsiloxane and polydiphenylsiloxane.

Low molecular weight functional polysiloxanes can also be employed, and are represented by the formula  $\text{R}_3\text{SiO}(\text{RQSIO})_y\text{SiR}_3$  where Q in said formula is a functional group. Examples of such functional polysiloxanes containing functional groups represented by Q are acrylamide functional siloxane fluids, acrylate functional siloxane fluids, amide functional siloxane fluids, amino functional siloxane fluids, carbinol functional siloxane fluids, carboxy functional siloxane fluids, chloroalkyl functional siloxane fluids, epoxy functional siloxane fluids, glycol functional siloxane fluids, ketal functional siloxane fluids, mercapto functional siloxane fluids, methyl ester functional siloxane fluids, perfluoro functional siloxane fluids, and silanol functional siloxanes.

This invention is not limited to swelling silicone elastomers with only low molecular weight polysiloxanes. Other types of solvents, such as organic compounds, can swell the silicone elastomer. Thus, a single solvent or a mixture of solvents may be used.

In general, the organic compounds are aromatic hydrocarbons, aliphatic hydrocarbons, alcohols, aldehydes, ketones, amines, esters, ethers, glycols, glycol ethers, alkyl halides or aromatic halides. Representative of some common organic solvents are alcohols such as methanol, ethanol, 1-propanol, cyclohexanol, benzyl alcohol, 2-octanol, ethylene glycol, propylene glycol and glycerol; aliphatic hydrocarbons such as pentane, cyclohexane, heptane, VM&P solvent and mineral spirits; alkyl halides such as chloroform, carbon tetrachloride, perchloroethylene, ethyl chloride and chlorobenzene; aromatic hydrocarbons such as benzene, toluene, ethylbenzene and xylene; esters such as ethyl acetate, isopropyl acetate, ethyl acetoacetate, amyl acetate, isobutyl isobutyrate and benzyl acetate; ethers such as ethyl ether, n-butyl ether, tetrahydrofuran and 1,4-dioxane; glycol ethers such as ethylene glycol monomethyl ether, ethylene glycol monomethyl ether acetate, diethylene glycol monobutyl ether and propylene glycol monophenyl ether; ketones such as acetone, methyl ethyl ketone, cyclohexanone, diacetone alcohol, methyl amyl ketone and diisobutyl ketone; petroleum hydrocarbons such as mineral oil, gasoline, naphtha, kerosene, gas oil, heavy oil and crude oil; lubricating

oils such as spindle oil and turbine oil; and fatty oils such as corn oil, soybean oil, olive oil, rape seed oil, cotton seed oil, sardine oil, herring oil and whale oil.

Other miscellaneous organic solvents can also be used, such as acetonitrile, nitromethane, dimethylformamide, propylene oxide, trioctyl phosphate, butyrolactone, furfural, pine oil, turpentine and m-cresol.

Further encompassed by the term solvent are volatile flavoring agents such as oil of wintergreen; peppermint oil; spearmint oil; menthol; vanilla; cinnamon oil; clove oil; bay oil; anise oil; eucalyptus oil; thyme oil; cedar leaf oil; oil of nutmeg; oil of sage; cassia oil; cocoa; licorice; high fructose corn syrup; citrus oils such as lemon, orange, lime and grapefruit; fruit essences such as apple, pear, peach, grape, strawberry, raspberry, cherry, plum, pineapple and apricot; and other useful flavoring agents including aldehydes and esters such as cinnamyl acetate, cinnamaldehyde, eugenyl formate, p-methylanisole, acetaldehyde, benzaldehyde, anisic aldehyde, citral, neral, decanal, vanillin, tolyl aldehyde, 2,6-dimethyloctanal and 2-ethyl butyraldehyde.

In addition, the term solvent is intended to include volatile fragrances such as natural products and perfume oils. Some representatives of typical natural products and perfume oils are ambergris, benzoin, civet, clove, leaf oil, jasmine, mate, mimosa, musk, myrrh, orris, sandalwood oil and vetiver oil; aroma chemicals such as amyl salicylate, amyl cinnamic aldehyde, benzyl acetate, citronellol, coumarin, geraniol, isobornyl acetate, ambrette, and terpinyl acetate; and the various classic family perfume oils such as the floral bouquet family, the oriental family, the chypre family, the woody family, the citrus family, the canoe family, the leather family, the spice family and the herbal family.

Our process is performed by simply combining in one pot the  $\text{=SiH}$  containing siloxane(s), the mono-alkenyl polyether, the alpha, omega-diene, the low molecular weight siloxane or solvent, and the platinum catalyst; and by then mixing these ingredients at room temperature until an elastomer, paste, or emulsion, is formed. If desired the elastomer, paste, or emulsion can be further diluted with an additional similar or dissimilar solvent(s), to form a final composition. A blend of hexane and tetrahydrofuran, a fragrance, an oil, or another low molecular weight siloxane, are examples of diluents that are so employed. Higher temperatures to speed up the process can also be used.

Additional amounts of low molecular weight siloxane or solvent are added to the gel, i.e., Step 3, and the resulting mixture is subjected to shear force to form the paste. In Step 4, shear force is again used, during or after water is added to the paste, to form the emulsion. Any type of mixing and shearing equipment may be used to perform these steps such as a batch mixer, planetary mixer, single or multiple screw extruder, dynamic or static mixer, colloid mill, homogenizer, sonolator or any combination thereof.

Typically, the process is conducted by using approximately a 1:1 molar ratio of the  $\text{=SiH}$  in the  $\text{=Si-H}$  containing siloxane and the vinyl in the alpha, omega-diene.

It is expected that useful materials may also be prepared by carrying out our process with an excess of either the  $\text{=Si-H}$  containing siloxane or the alpha, omega-diene, but this would be considered a less efficient use of the materials. The remainder of our claimed composition comprises the low molecular weight siloxane or solvent, in amounts generally within the range of 65-98 percent by weight of the composition, but preferably 80-98 percent by weight.

The silicone elastomer, silicone gel, silicone paste and silicone emulsion compositions of this invention have particular value in the personal care arena. Because of the unique volatility characteristics of the VMS component of the preferred compositions, they can be used alone, or blended with other cosmetic fluids, to form a variety of over-the-counter (OTC) personal care products.

Thus, they are useful as carriers in antiperspirants and deodorants, since they leave a dry feel, and do not cool the skin upon evaporation. They are lubricious and will improve the properties of skin creams, skin care lotions, moisturizers, facial treatments such as acne or wrinkle removers, personal and facial cleansers, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave and after-shave lotions, liquid soaps, shaving soaps and shaving lathers. They can be used in hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories and cuticle coats, to enhance gloss and drying time, and provide conditioning benefits.

In cosmetics, they will readily function as leveling and spreading agents for pigments in make-ups, color cosmetics, foundations, blushes, lipsticks, lip balms, eyeliners, mascaras, oil removers, color cosmetic removers, and powders. They are also useful as delivery systems for oil and water soluble substances such as vitamins. When incorporated into sticks, gels, lotions, aerosols and roll-ons, the compositions impart a dry, silky-smooth, payout.

In addition, the claimed compositions exhibit a variety of advantageous and beneficial properties such as clarity, shelf stability, and ease of preparation. Hence, they have wide application, but especially in antiperspirants, deodorants, in perfumes as a carrier and for conditioning hair.

The silicone elastomers, gels, pastes and emulsions, have uses beyond the personal care arena, including their use as a filler or insulation material for electrical cable, a soil or water barrier for in-ground stabilization or as a replacement for epoxy materials used in coil-on-plug designs in the electronics industry.

They are also useful as carrier for crosslinked silicone rubber particles. In that application, (i) they allow ease of incorporation of the particles into such silicone or organic phases as seal ants, paints, coatings, greases, adhesives, antifoams, and potting compounds; and (ii) they provide for modifying rheological, physical, or energy absorbing prop-

erties of such phases in either their neat or finished condition.

In addition, our silicone elastomers, gels, pastes and emulsions are capable of functioning as carriers for pharmaceuticals, biocides, herbicides, pesticides, and other biologically active substances; and can be used to incorporate water and water-soluble substances into hydrophobic systems. Examples of some water-soluble substances are salicylic acid, glycerol, enzymes and glycolic acid.

The following example is set forth for the purpose of further illustrating the one pot method according to the present invention.

#### Example 1 - One Pot Method

15 g of an organopolysiloxane with the average structure  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_{93}(\text{MeHSiO})_6\text{SiMe}_3$ , 0.53 g of a polyether with the average structure  $\text{CH}_2=\text{CHCH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_7\text{C}(\text{O})\text{CH}_3$ , 72 g decamethylcyclopentasiloxane, 0.48 g 1,5-hexadiene, and 40  $\mu\text{l}$  Karstedt catalyst (i.e., two weight percent platinum in decamethylcyclopentasiloxane), were first placed in a reaction vessel. Then, the solution was heated in a 60°C. bath and vigorously stirred by a large magnetic stirring bar. Gelation occurred quickly, and the gel was heated in the 60°C. bath for 5 hours. A slightly hazy gel resulted, and 70 g of the gel was sheared and swollen with 55 g of decamethylcyclopentasiloxane. A uniform paste was obtained having a viscosity at a shear rate of 0.01  $\text{s}^{-1}$  of  $1.17 \times 10^6$  cP (mPa · s). Twenty grams of the uniform paste and 15 g of deionized water were mixed in a glass jar with a mechanical stirrer, forming a white emulsion paste having a viscosity at a shear rate of 0.01  $\text{s}^{-1}$  of  $5.29 \times 10^6$  cP (mPa · s). No surfactant was required to make this emulsion.

#### Claims

1. A method of making a silicone elastomer comprising combining and reacting:

(A) a  $\text{=Si-H}$  containing polysiloxane of the formula  $\text{R}_3\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_3$  or the formula  $(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b$ , and optionally an  $\text{=Si-H}$  containing polysiloxane of the formula  $\text{NR}_2\text{SiO}(\text{R}'_2\text{SiO})_c\text{SiR}_2\text{H}$  or an  $\text{=Si-H}$  containing polysiloxane of the formula  $\text{HR}_2\text{SiO}(\text{R}'_2\text{SiO})_a(\text{R}''\text{HSiO})_b\text{SiR}_2\text{H}$ , where R, R', and R'' are alkyl groups with 1-6 carbon atoms, a is 0-250, b is 1-250, and c is 0-250;

(B) an unsaturated hydrocarbon selected from the group consisting of alpha, omega-dienes of the formula  $\text{CH}_2=\text{CH}(\text{CH}_2)_x\text{CH}=\text{CH}_2$ , alpha, omega-diynes of the formula  $\text{CH}=\text{C}(\text{CH}_2)_x\text{C}=\text{CH}$ , and alpha, omega-ene-yne of the formula  $\text{CH}_2=\text{CH}(\text{CH}_2)_x\text{C}=\text{CH}$ , where x is 1-20;

(C) a platinum catalyst;

(D) a mono-alkenyl polyether of the formula  $\text{CH}_2=\text{CH}(\text{CH}_2)_x\text{O}(\text{CH}_2\text{CH}_2\text{O})_y(\text{CH}_2\text{CH}_2\text{CHO})_z\text{T}$ , or the formula  $\text{CH}_2=\text{CH-Q-O}(\text{CH}_2\text{CH}_2\text{O})_y(\text{CH}_2\text{CH}_2\text{CHO})_z\text{T}$ , where T is hydrogen, a  $\text{C}_1$ - $\text{C}_{10}$  alkyl group, an aryl group, or a  $\text{C}_1$ - $\text{C}_{20}$  acyl group; Q is a divalent linking group containing unsaturation; x is 1-6, y is zero or 1-100; and z is zero or 1-100; provided y and z are both not zero; and

(E) a solvent selected from the group consisting of (i) organic compounds, (ii) compounds containing a silicon atom, (iii) mixtures of organic compounds, (iv) mixtures of compounds containing a silicon atom, and (v) mixtures of organic compounds and compounds containing a silicon atom.

2. A method according to claim 1 including the further steps of adding additional amounts of solvent(s) to the silicone elastomer, and shearing the solvent(s) and silicone elastomer until a silicone paste is formed.

3. A method according to claim 2 including the further steps of adding water to the silicone paste, and shearing the water and silicone paste until a silicone emulsion is formed.

4. A method according to claim 3 in which the silicone emulsion is formed free of the presence of a surfactant.

5. A method according to claim 1 in which the solvent is a linear volatile methyl siloxane of the formula  $(\text{CH}_3)_3\text{SiO}\{(\text{CH}_3)_2\text{SiO}\}_y\text{Si}(\text{CH}_3)_3$  where y is 0-5, or a cyclic volatile methyl siloxane of the formula  $\{(\text{CH}_3)_2\text{SiO}\}_z$  where z is 3-8, the volatile methyl siloxane have a boiling point less than 250°C. and a viscosity of 0.65-5.0 centistokes ( $\text{mm}^2/\text{s}$ ).

6. A silicone elastomer obtainable by the method of claim 1.

7. A silicone paste obtainable by the method of claim 2.

8. A silicone emulsion obtainable by the method of claim 3.

9. Use of the silicone elastomer of claim 6 in personal care applications by applying to the hair, skin or underarm.

10. Use of the silicone paste of claim 7 in personal care applications by applying to the hair, skin or underarm.

5 11. Use of the silicone emulsion of claim 8 in personal care applications by applying to the hair, skin or underarm.

12. A method of providing a barrier film to the surface of a substrate comprising applying to the substrate the silicone elastomer of claim 6 and allowing the solvent to evaporate.

10 13. A method of providing a barrier film to the surface of a substrate comprising applying to the substrate the silicone paste of claim 7 and allowing the solvent to evaporate.

14. A method of providing a barrier film to the surface of a substrate comprising applying to the substrate the silicone emulsion of claim 8 and allowing the solvent to evaporate.

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# EUROPEAN SEARCH REPORT

Application Number  
EP 98 10 9954

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	EP 0 298 402 A (DOW) 11 January 1989 * page 2, line 1 - line 2 * * page 2, line 24 - line 25 * * page 3, line 48 - line 49 * * example 1 * * claim 1 * ---	1,6,8	C08G77/38 C08G77/46 C08G77/50
Y	EP 0 501 791 A (SHIN-ETSU) 2 September 1992 * abstract * * page 4, line 50 - page 5, line 30 * * examples 1,2 * * page 8, line 1 - page 9, line 2 * ---	1-14	
Y	GB 1 093 904 A (DOW) * claims 1-5 * ---	1-14	
Y	US 5 493 041 A (T. N. BIGGS ET AL.) 20 February 1996 * column 3, line 32 - line 40 * * claim 1 * -----	1-14	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
			C08G
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 23 July 1998	Examiner Hoepfner, W
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

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